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IS 11959 (1987): Method for metallographic determination of microstructure in hardmetals [MTD 25: Powder Metallurgical Materials and Products]

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METHOD FOR METALLOGRAPHIC DETERMINATION OF MICROSTRUCTURE IN HARDMETALS

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NEW DELHI 110002

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Indian Standard

METHOD FOR METALLOGRAPHIC DETERMINATION OF MICROSTRUCTURE IN HARDMETALS

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Indian Standard

METHOD FOR METALLOGRAPHIC DETERMINATION OF MICROSTRUCTURE IN HARDMETALS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 February 1987, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 With the increasing use of hardmetals, a need has been felt for having standards on the metallographic examination. This standard is one of the series on this subject. It is hoped that the formulation of this standard will be of considerable use to the industry.

0.3 In preparation of this standard, assistance has been derived from the following standards:

ISO 4499-1978 Hardmetals — Metallographic determination of microstructure. International Organization for Standardization (ISO).

ASTM B 657-79 Method for metallographic determination of microstructure in cemented carbides. American Society of Testing and Materials.

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard specifies the methods of metallographic determination of the microstructure of hardmetals.

2. DEFINITION OF SYMBOLS

2.1

Symbol	*Definition*
α-phase	Tungsten carbide
β-phase	Binder phase (for example, Co, Ni, Fe)
γ-phase	Carbide of a cubic lattice (for example, TiC, TaC, NbC) that may contain other carbides (for example, WC) in solid solution
η-type phases	Multiple carbides of tungsten and at least one metal of the binder phase

3. APPARATUS

3.1 Metallographic Microscope — capable of magnifications up to 1 500 ×.

3.2 Equipment — for the metallographic preparation of test specimen.

4. SPECIMEN PREPARATION

4.1 Test specimen shall be selected and prepared for metallographic examination as per the procedure given in IS : 11520-1985*.

4.2 The surface to be examined shall be free from grinding and polishing mark. Care shall be taken to avoid tearing out of particles, which may lead to a wrong evaluation of microstructure.

5. PROCEDURE

5.1 Examine the microstructure by gradual development of the phases by etching. Examples of suitable etching techniques are given in Table 1. Care shall be taken to ensure that the true microstruture is revealed.

5.2 Determine the presence of η-type phases by lightly etching the section with Technique 1 (*see* Table 1). Phases of η-type are coloured orange to brown. γ-phase may etch lightly, while the other phases remain unetched. Etching by Technique 1 does not prevent subsequent etching by Techniques 2 or 3. Examine the entire section at low magnification and, if necessary, at magnification up to 1 500 ×. Note and record the existence of η-type phases and their distribution.

5.3 Determine the presence of γ-phases by etching with Technique 2 (*see* Table 1). This phase appears light yellowish brown and has a typically rounded shape (*see* Fig. 1). Examine the etched section and note and record the existence of γ-phase. Estimate and record its size according to Fig. 1 as γ-fine, γ-medium, or γ-coarse.

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*Method for metallographic sample preparation of hardmetals.

5.4 Determine the presence of α-phase by etching the surface with Technique 3, or in case γ-phase is present, with Technique 2. The α-phase appears grey and often has an angular shape. Examine the etched section and note and record the presence of an α-phase. Estimate and record its size according to Fig. 2 as α-fine, α-medium, or α-coarse.

5.5 Identify the β-phase after etching the surface by Technique 2 or 3 (*see* Table 1). This phase remains white.

6. TEST REPORT

6.1 The test report shall include the following information:

a) Reference to this standard,
b) Complete identification of the test sample,
c) The results obtained, and
d) Details of any occurrence which may have affected the result.

TABLE 1 ETCHING TECHNIQUE

Etching Technique	Composition of Etchants	Conditions of Etching	Application of Etching
1	SOLUTION A — Freshly prepared solution of equal quantities of 10 to 20 percent (*m/m*) aqueous solutions of potassium hexacyanoferrate (III) (potassium ferricyanide) and potassium or sodium hydroxide	Etch in solution A at approximately 20°C for 2 to 20 s. Flush the test-piece section with water immediately, without removing the oxide layer. Dry the surface carefully with acetone or alcohol without wiping	Identification of η-phases
2	SOLUTION A — *See* Technique 1 SOLUTION B — A solution of equal volumes of concentrated hydrochloric acid and water	Etch at approximately 20°C in solution A for 3 to 5 min. Then wash in water and etch in solution B for approximaely 10 s. Next wash in water, then in alcohol and dry the test-piece section. Finally, etch in solution A for approximately 20 s	Identification of γ-phase
3	SOLUTION A — *See* Technique 1	Etch in solution A at approximately 20°C for 3 to 6 min	Identification of α-phase

NOTE — The separate solutions of potassium hexacyanoferrate (III) and potassium or sodium hydroxide may be stored for a long time, but must be freshly mixed each day when used.

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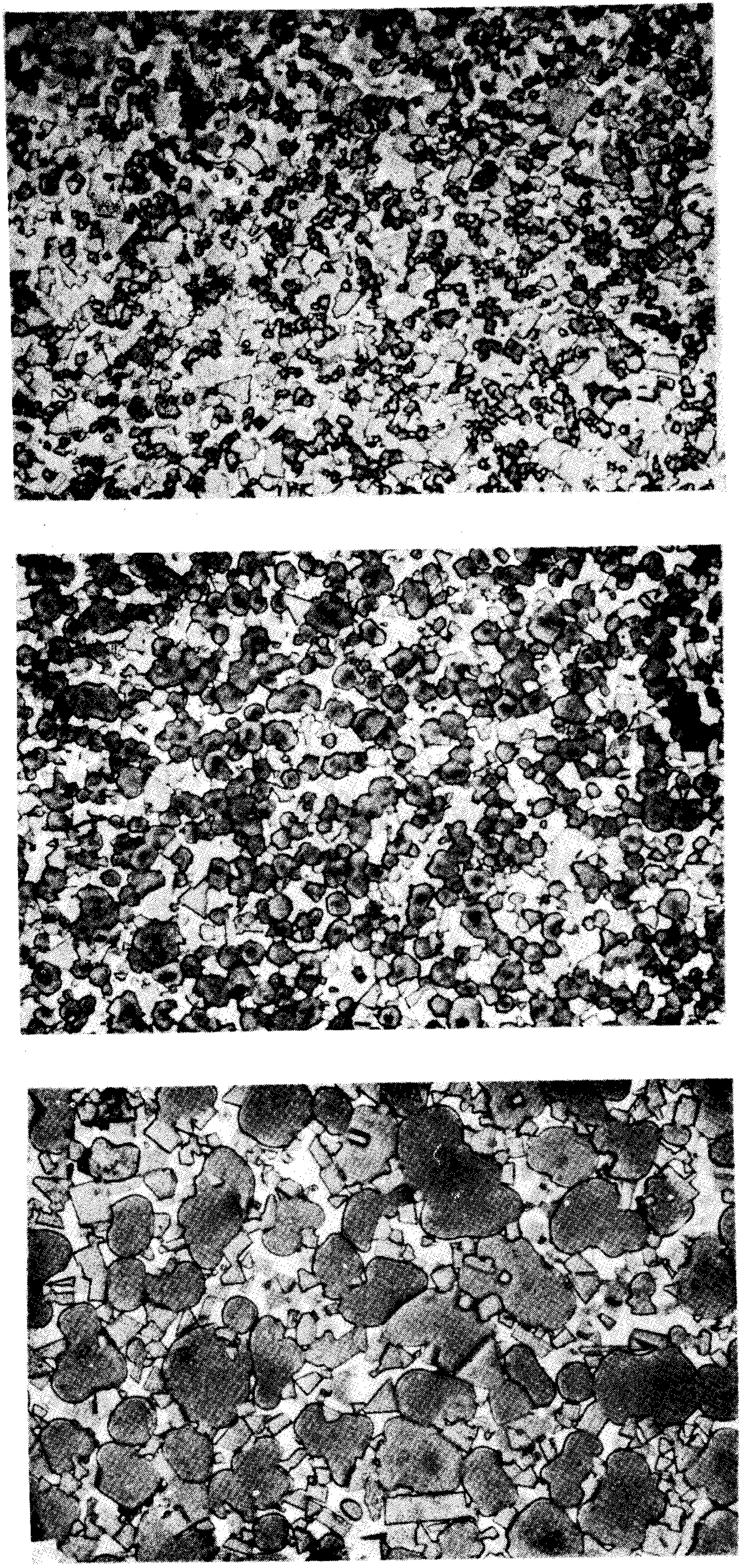


γ-fine

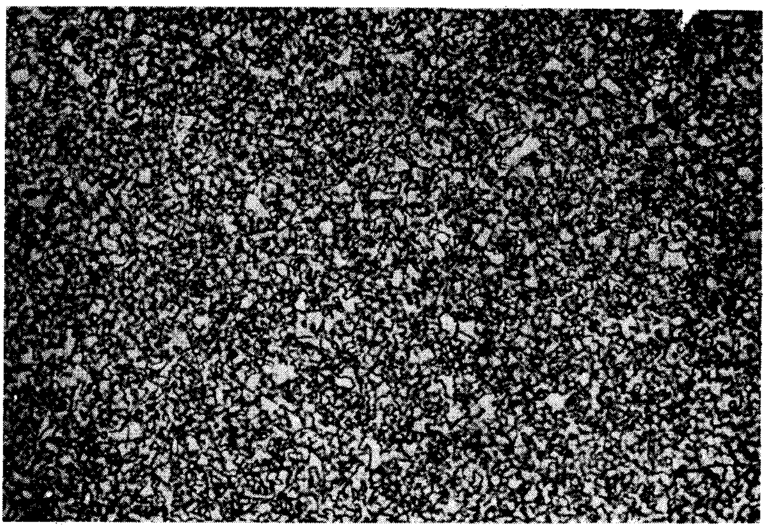
γ-medium

γ-coarse

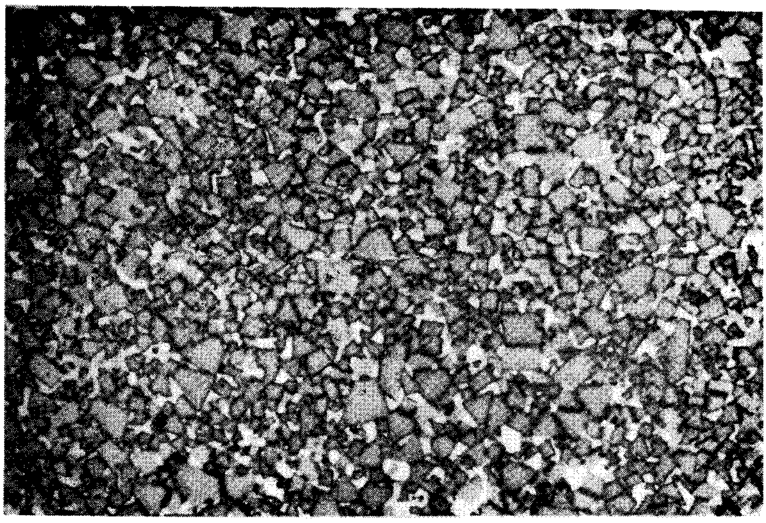
FIGURE 1 — γ-phase × 1 500

This figure is reproduced from ISO 4499.

α-fine



α-medium



α-coarse

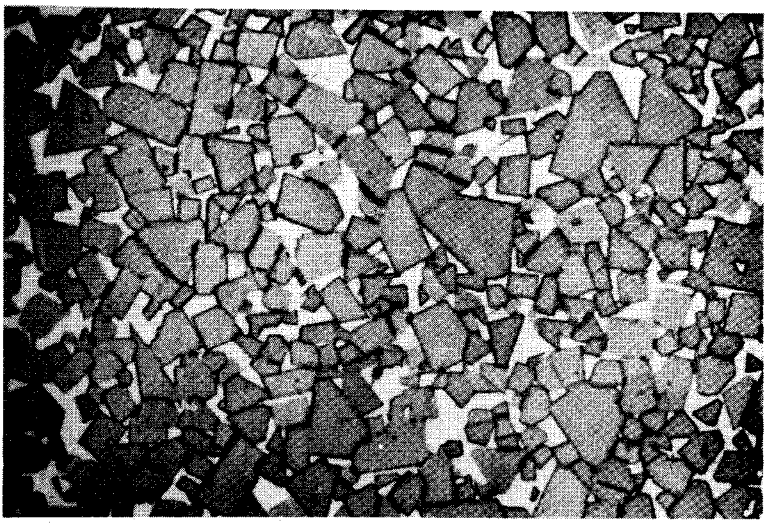


FIGURE 2 – α-**phase** × **1 500**

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